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1-(2,3-Dicyanophenyl)pyridin-1-ium-4-olate monohydrate

Zhe Han,[a,b] Ying Zhu,[b] Xuelei Tian[a] and Suping Feng[c]*

[a]School of Materials Science and Engineering of Shandong University, 250014 Jinan, People's Republic of China, [b]New Materials Institute of Shandong Academy of Sciences, 250014 Jinan, People's Republic of China, and [c]School of Environmental Science and Engineering of Shandong University, 250100 Jinan, People's Republic of China
Correspondence e-mail: fengsuping@126.com

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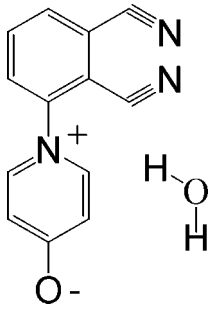
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.044; wR factor = 0.124; data-to-parameter ratio = 12.3.

In the crystal structure of the title compound, $C_{13}H_7N_3O\cdot H_2O$, the components are associated into chains along [010] through strong O—H···O hydrogen bonds with the free water molecules as bridging ligands. These chains are further cross-linked by C—H···O and C—H···N hydrogen bonds, forming a three-dimensional structure.

Related literature

For the preparation of the title compound, see: Archibald *et al.* (1994)



Experimental

Crystal data

$C_{13}H_7N_3O\cdot H_2O$ M_r = 239.23
Monoclinic, $P2_1/c$
a = 11.977 (2) Å
b = 7.2497 (12) Å
c = 13.850 (2) Å
β = 94.244 (3)°
V = 1199.3 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 K
0.12 × 0.10 × 0.08 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.989, T_{max} = 0.993
5795 measured reflections
2112 independent reflections
1349 reflections with $I > 2\sigma(I)$
R_{int} = 0.042

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.124
S = 1.03
2112 reflections
172 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.12 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1W—H1WA···O1	0.98 (3)	1.79 (3)	2.763 (2)	170 (3)
O1W—H1WB···O1^{i}	0.87 (3)	1.88 (3)	2.721 (2)	162 (3)
C6—H6A···O1W^{ii}	0.93	2.37	3.302 (3)	177
C13—H13A···O1W^{iii}	0.93	2.38	3.311 (3)	175
C9—H9A···N1^{iv}	0.93	2.55	3.440 (2)	160

Symmetry codes: (i) $-x+1, y-\frac{1}{2}, -z+\frac{3}{2}$; (ii) $-x+1, -y+1, -z+1$; (iii) $-x+1, -y, -z+1$; (iv) $x, y+1, z$.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BV2216).

References

Archibald, S. J., Blake, A. J., Schroder, M. & Winpenny, R. E. P. (1994). *Chem. Commun.* pp. 1669—1670.
Bruker (2001). *SAINT-Plus* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2004). *APEX2*. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supplementary materials

Acta Cryst. (2013). E**69**, o767 [doi:10.1107/S1600536813008891]

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Comment

In the crystal structure of the title compound, $(C_{13}H_7N_3O)(H_2O)$, the units are associated into a chain through strong O—H···O hydrogen bonds with the free water molecules as the bridging ligands. These chains are further crosslinked by C-H···O interactions. In addition there are weak pairwise C—H···N hydrogen bonds.

Experimental

The preparation of the title compound, see, Archibald *et al.* (1994)

Refinement

All hydrogen atoms bound to carbon were refined using a riding model with distance C—H = 0.93 Å, U_{iso} = 1.2U_{eq} (C) for aromatic atoms. The H atoms of the water molecule were located from difference density maps and were refined isotrpically.

Computing details

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus* (Bruker, 2001); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

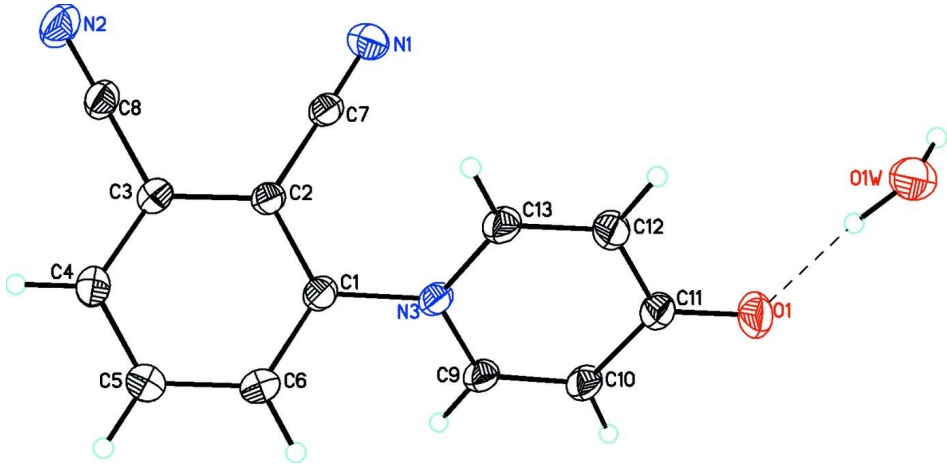

Figure 1

The asymmetric unit of the title compound with the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level; H atoms are given as spheres of arbitrary radius.

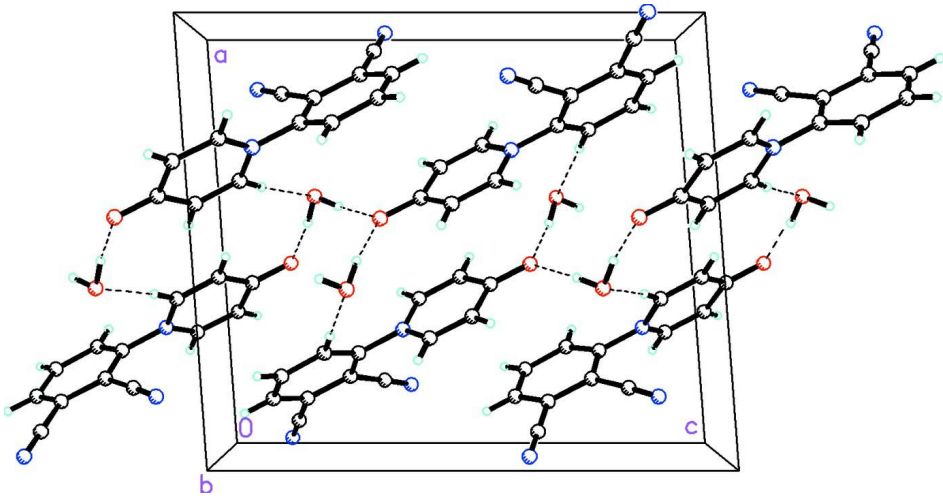

Figure 2

The packing diagram of the title compound with hydrogen bonds shown as dashed lines.

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$C_{13}H_7N_3O·H_2O$
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$a = 11.977$ (2) Å
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$c = 13.850$ (2) Å
$\beta = 94.244$ (3)°
$V = 1199.3$ (3) Å^3
$Z = 4$
$F(000) = 496$
$D_x = 1.325$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2117 reflections
$\theta = 1.7$–25.0°
$\mu = 0.09$ mm^{-1}
$T = 293$ K
Block, colorless
0.12 × 0.10 × 0.08 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
$T_{min} = 0.989$, $T_{max} = 0.993$
5795 measured reflections
2112 independent reflections
1349 reflections with $I > 2\sigma(I)$
$R_{int} = 0.042$
$\theta_{max} = 25.0°$, $\theta_{min} = 1.7°$
$h = -14\rightarrow14$
$k = 0\rightarrow8$
$l = 0\rightarrow16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.124$
$S = 1.03$
2112 reflections
172 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0572P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.12$ e Å^{-3}

$\Delta\rho_{min}$ = −0.14 e Å^{-3}

Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.015 (4)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.44687 (15)	0.4052 (2)	0.64729 (11)	0.0821 (6)
N1	0.14211 (16)	−0.0800 (2)	0.38040 (14)	0.0686 (6)
N2	0.00645 (18)	−0.1230 (3)	0.11391 (13)	0.0794 (7)
N3	0.29064 (13)	0.35472 (19)	0.37786 (11)	0.0477 (5)
C1	0.23465 (16)	0.3377 (2)	0.28309 (13)	0.0469 (5)
C2	0.17290 (15)	0.1798 (2)	0.25806 (13)	0.0437 (5)
C3	0.11900 (16)	0.1688 (2)	0.16464 (14)	0.0479 (5)
C4	0.12482 (18)	0.3119 (3)	0.09995 (14)	0.0578 (6)
H4A	0.0889	0.3032	0.0383	0.069*
C5	0.1844 (2)	0.4680 (3)	0.12749 (15)	0.0652 (7)
H5A	0.1875	0.5659	0.0844	0.078*
C6	0.23959 (19)	0.4810 (3)	0.21802 (16)	0.0609 (6)
H6A	0.2803	0.5867	0.2354	0.073*
C7	0.15735 (17)	0.0352 (3)	0.32680 (15)	0.0482 (5)
C8	0.05625 (18)	0.0052 (3)	0.13666 (14)	0.0561 (6)
C9	0.27488 (17)	0.5072 (2)	0.43253 (15)	0.0536 (6)
H9A	0.2273	0.5997	0.4075	0.064*
C10	0.32575 (18)	0.5283 (3)	0.52132 (15)	0.0567 (6)
H10A	0.3128	0.6349	0.5561	0.068*
C11	0.39919 (18)	0.3909 (3)	0.56354 (15)	0.0578 (6)
C12	0.41474 (18)	0.2360 (3)	0.50307 (15)	0.0618 (6)
H12A	0.4627	0.1421	0.5257	0.074*
C13	0.36240 (17)	0.2209 (3)	0.41422 (14)	0.0570 (6)
H13A	0.3752	0.1176	0.3768	0.068*
O1W	0.60702 (15)	0.1549 (2)	0.71708 (14)	0.0732 (5)
H1WA	0.544 (2)	0.236 (4)	0.6961 (19)	0.121 (11)*
H1WB	0.583 (2)	0.095 (4)	0.766 (2)	0.120 (12)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.1059 (14)	0.0738 (11)	0.0615 (10)	0.0203 (9)	−0.0291 (9)	−0.0165 (8)
N1	0.0756 (14)	0.0545 (11)	0.0750 (14)	−0.0007 (9)	0.0007 (11)	0.0099 (10)
N2	0.0927 (16)	0.0754 (13)	0.0685 (14)	−0.0333 (12)	−0.0050 (11)	−0.0101 (10)

N3	0.0482 (10)	0.0384 (9)	0.0549 (10)	0.0008 (7)	−0.0071 (8)	−0.0087 (7)
C1	0.0472 (12)	0.0433 (11)	0.0494 (12)	−0.0008 (9)	−0.0022 (9)	−0.0062 (9)
C2	0.0458 (12)	0.0385 (10)	0.0467 (11)	−0.0013 (8)	0.0019 (9)	−0.0029 (8)
C3	0.0481 (12)	0.0464 (11)	0.0492 (12)	−0.0050 (9)	0.0033 (9)	−0.0060 (9)
C4	0.0663 (15)	0.0606 (13)	0.0452 (12)	−0.0070 (11)	−0.0046 (10)	−0.0017 (10)
C5	0.0849 (17)	0.0540 (13)	0.0557 (14)	−0.0102 (12)	−0.0026 (12)	0.0070 (10)
C6	0.0718 (16)	0.0459 (12)	0.0637 (15)	−0.0143 (11)	−0.0042 (12)	−0.0005 (10)
C7	0.0487 (13)	0.0419 (11)	0.0531 (13)	−0.0006 (9)	−0.0016 (10)	−0.0078 (10)
C8	0.0590 (14)	0.0586 (13)	0.0497 (13)	−0.0132 (11)	−0.0019 (10)	−0.0053 (10)
C9	0.0571 (14)	0.0377 (11)	0.0640 (14)	0.0032 (9)	−0.0082 (11)	−0.0077 (9)
C10	0.0642 (14)	0.0435 (11)	0.0608 (14)	0.0065 (10)	−0.0062 (11)	−0.0141 (9)
C11	0.0654 (15)	0.0504 (12)	0.0555 (14)	0.0030 (11)	−0.0088 (11)	−0.0090 (10)
C12	0.0653 (16)	0.0503 (12)	0.0667 (15)	0.0159 (10)	−0.0148 (11)	−0.0088 (10)
C13	0.0585 (14)	0.0434 (11)	0.0673 (14)	0.0088 (10)	−0.0082 (11)	−0.0128 (10)
O1W	0.0750 (13)	0.0633 (11)	0.0813 (13)	0.0009 (9)	0.0063 (10)	0.0050 (9)

Geometric parameters (Å, º)

O1—C11	1.259 (2)	C5—C6	1.376 (3)
N1—C7	1.141 (2)	C5—H5A	0.9300
N2—C8	1.136 (2)	C6—H6A	0.9300
N3—C9	1.361 (2)	C9—C10	1.340 (3)
N3—C13	1.367 (2)	C9—H9A	0.9300
N3—C1	1.434 (2)	C10—C11	1.425 (3)
C1—C6	1.379 (3)	C10—H10A	0.9300
C1—C2	1.393 (2)	C11—C12	1.421 (3)
C2—C3	1.405 (2)	C12—C13	1.343 (2)
C2—C7	1.437 (3)	C12—H12A	0.9300
C3—C4	1.375 (3)	C13—H13A	0.9300
C3—C8	1.442 (3)	O1W—H1WA	0.98 (3)
C4—C5	1.377 (3)	O1W—H1WB	0.87 (3)
C4—H4A	0.9300		
C9—N3—C13	118.73 (16)	C1—C6—H6A	119.9
C9—N3—C1	120.28 (15)	N1—C7—C2	178.2 (2)
C13—N3—C1	120.98 (15)	N2—C8—C3	179.4 (2)
C6—C1—C2	120.27 (17)	C10—C9—N3	122.07 (18)
C6—C1—N3	119.57 (16)	C10—C9—H9A	119.0
C2—C1—N3	120.14 (16)	N3—C9—H9A	119.0
C1—C2—C3	118.35 (16)	C9—C10—C11	121.43 (18)
C1—C2—C7	121.80 (16)	C9—C10—H10A	119.3
C3—C2—C7	119.73 (16)	C11—C10—H10A	119.3
C4—C3—C2	121.03 (17)	O1—C11—C12	122.52 (18)
C4—C3—C8	119.71 (17)	O1—C11—C10	123.01 (18)
C2—C3—C8	119.26 (17)	C12—C11—C10	114.47 (18)
C3—C4—C5	119.31 (18)	C13—C12—C11	122.10 (18)
C3—C4—H4A	120.3	C13—C12—H12A	119.0
C5—C4—H4A	120.3	C11—C12—H12A	119.0
C6—C5—C4	120.84 (19)	C12—C13—N3	121.17 (17)
C6—C5—H5A	119.6	C12—C13—H13A	119.4

C4—C5—H5A	119.6	N3—C13—H13A	119.4
C5—C6—C1	120.18 (18)	H1WA—O1W—H1WB	104 (3)
C5—C6—H6A	119.9		

C9—N3—C1—C6	50.0 (3)	C2—C1—C6—C5	−0.7 (3)
C13—N3—C1—C6	−128.6 (2)	N3—C1—C6—C5	−178.7 (2)
C9—N3—C1—C2	−128.0 (2)	C1—C2—C7—N1	140 (7)
C13—N3—C1—C2	53.4 (3)	C3—C2—C7—N1	−36 (7)
C6—C1—C2—C3	1.7 (3)	C4—C3—C8—N2	−9 (28)
N3—C1—C2—C3	179.77 (17)	C2—C3—C8—N2	171 (100)
C6—C1—C2—C7	−174.18 (19)	C13—N3—C9—C10	−1.3 (3)
N3—C1—C2—C7	3.9 (3)	C1—N3—C9—C10	−179.9 (2)
C1—C2—C3—C4	−1.4 (3)	N3—C9—C10—C11	−0.2 (3)
C7—C2—C3—C4	174.61 (19)	C9—C10—C11—O1	−178.9 (2)
C1—C2—C3—C8	178.93 (18)	C9—C10—C11—C12	1.4 (3)
C7—C2—C3—C8	−5.1 (3)	O1—C11—C12—C13	179.2 (2)
C2—C3—C4—C5	0.0 (3)	C10—C11—C12—C13	−1.1 (3)
C8—C3—C4—C5	179.7 (2)	C11—C12—C13—N3	−0.4 (3)
C3—C4—C5—C6	1.1 (3)	C9—N3—C13—C12	1.6 (3)
C4—C5—C6—C1	−0.8 (3)	C1—N3—C13—C12	−179.8 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H1*WA*···O1	0.98 (3)	1.79 (3)	2.763 (2)	170 (3)
O1*W*—H1*WB*···O1^{i}	0.87 (3)	1.88 (3)	2.721 (2)	162 (3)
C6—H6*A*···O1*W*^{ii}	0.93	2.37	3.302 (3)	177
C13—H13*A*···O1*W*^{iii}	0.93	2.38	3.311 (3)	175
C9—H9*A*···N1^{iv}	0.93	2.55	3.440 (2)	160

Symmetry codes: (i) −*x*+1, *y*−1/2, −*z*+3/2; (ii) −*x*+1, −*y*+1, −*z*+1; (iii) −*x*+1, −*y*, −*z*+1; (iv) *x*, *y*+1, *z*.